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HIGH TEMPERATURE OXIDATION PROTECTIVE COATINGS FOR VANADIUM-BASE ALLOYS

Contract N600(19) 59182

Department of the Navy
Bureau of Naval Weapons
Washington 25, D. C.
Attention: Code RRMA-222

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ARMOUR RESEARCH FOUNDATION OF ILLINOIS INSTITUTE OF TECHNOLOGY

#1.60

HIGH TEMPERATURE

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FOR VANADIUM-BASE ALLOYS

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ARMOUR RESEARCH FOUNDATION
of
ILLINOIS INSTITUTE OF TECHNOLOGY
Technology Center
Chicago 16, Illinois

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OXIDATION PROTECTIVE COATINGS
FOR VANADIUM-BASE ALLOYS

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June 7, 1963

HIGH TEMPERATURE OXIDATION PROTECTIVE COATINGS FOR VANADIUM-BASE ALLOYS

ABSTRACT

The influence of thermal history for a standardized pack siliconizing process has been established for 0.020- to 0.030-inch sheet of the V-60w/o Cb-1w/o Ti alloy. Studies of coating thickness as a function of siliconizing time over the temperature range of 2000 to 2200°F ~~have~~ were ~~been~~ made. Coating thickness of 2.25 to 2.75 mils appears to offer optimum oxidation protection based on static air oxidation tests at 2200°F; specimens were ~~have been~~ exposed for more than 500 hours without evidence of failure. Thick coatings, ~~i.e.~~ (4 mils or greater) suffer gross delamination or severe cracking during elastic bending of sheet materials which results in a loss of protective capability. The thicker coatings also appear to behave poorly in high temperature, thermal cycle oxidation tests in an oxygen-hydrogen torch.

HIGH TEMPERATURE OXIDATION PROTECTIVE COATINGS FOR VANADIUM-BASE ALLOYS

I. INTRODUCTION

This is the fourth bimonthly progress report under Contract N600 (19) 59182, summarizing the work performed on ARF Project B6001 during the period from March 13, 1963 to May 12, 1963.

Efforts are being devoted to optimizing the highly promising silicide coatings for vanadium-columbium alloys. The influence of variations in the pack siliconizing process on static oxidation life and coating adherence during both elastic and plastic deformation is being investigated. The reliability of this coating on several of the higher-strength alloys has been studied and will be evaluated in more detail after the study of coating process variables has been completed.

To date, two of the most promising vanadium-columbium alloys are V-60Cb-1Ti and V-20Cb-4Ti-1Zr-0.075C* and 100-pound ingots of these compositions have been fabricated to sheet under Contract NOw 62-0101-c, "Pilot Evaluation of Vanadium Alloys." Specimens of these alloys are being coated for evaluation by aerospace and other organizations participating in the data-exchange program under the above contract.

Other compositions studied include additions of tantalum, hafnium, tungsten, titanium, molybdenum, and zirconium in varying amounts. Fifteen of these alloys have been siliconized and oxidation tested, with a minimum life, established at 2200°F, of 150 hours. The most promising of these alloys from the standpoint of mechanical properties was V-60Cb-1Zr-0.1C; a 100-pound ingot of this composition is currently being prepared for evaluation at the Foundation and by the organiziations requesting material under the pilot evaluation program.

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*Compositions are reported in weight per cent.

II. EXPERIMENTAL RESULTS

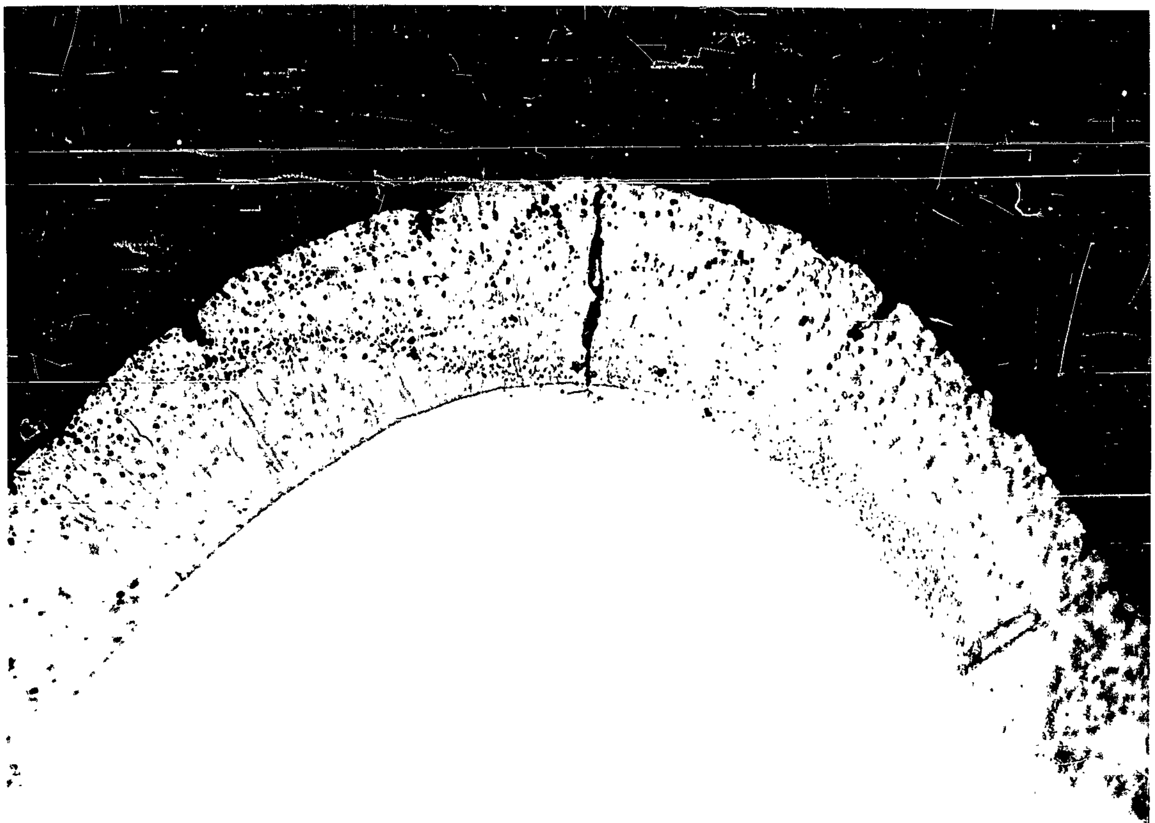
A. A Study of Pack Siliconizing Process Variables

1. Introduction

Previous pack variable studies on 0.050 inch and thicker sheet materials were made to establish the parameters necessary for producing siliconized alloys having maximum oxidation resistance. Processing for 16 hours at 2200°F in small retorts using a 10:1 Si:NaF ratio with the activator in a separate container under a static argon atmosphere[1] was found to be optimum. However, when coating thin sheet (20-30 mils), or using large retorts, several problems were encountered. The pack sintering that occurs during a continuous 16 hour siliconizing treatment causes excessive warpage and distortion of the thin material. Furthermore, the thick coatings that are produced on thin stock have relatively poor adherence and fail by laminar separation during elastic bending. These factors have led to the investigation of processing variables adaptable to the coating of thin sheet.

In this study, pack and retort design, activator species, and atmosphere were kept constant while silicon powder size, edge and surface preparation techniques, activator concentration, time, and temperature were varied. In the previous bimonthly report, the results of variations in surface preparation procedures on coating performance were described. These results have shown that oxidation life is not dependent on edge and surface preparation as long as the edges are uniformly round and free from shear cuts introduced during sizing, and the surface is clean and free of oxide prior to being sealed in the pack retort. However, barrel tumbling produces the most uniform specimens, which have a generous radius of curvature at edges. Specimens prepared in this manner have a minimum of cracks or growth flaws at edges during subsequent coating. The edge of a 22 mil thick siliconized V-60Cb-1Ti alloy specimen prepared by barrel tumbling may be seen in Figure 1. A crack which extends from the surface to the innermost diffusion layer in a direction normal to the base metal-coating interface may

1. J. J. Rausch and F. C. Holtz, "High-Temperature Oxidation Protective Coatings for Vanadium-Base Alloys," Final Report, Contract NOw 61-0806-c. August, 1962.



Neg. No. 24918 X 500

Fig. 1

V-60Cb-1Ti pack siliconized for 2.75 hr. at 2150°F. Sample from Run 21.

be observed. In less generously rounded specimens the occurrence and density of such cracks is greater; however, this appears to have little influence on coating performance during static oxidation exposure.

In order to minimize pack sintering during the coating process, the particle size of the silicon powder used in the pack was increased from +200, -325 mesh to +35, -100 mesh. This appears to have no pronounced influence on coating performance; however, this will be checked in greater detail in later work.

2. Influence of Time and Temperature on Coating Thickness

Primary emphasis during this report period has been directed to studying the influence of time and temperature on coating oxidation life and behavior under stress. The thermal treatments employed during siliconizing appear in Table 1. During each of these runs, -35, +100 mesh solar cell grade silicon powder and NaF at a concentration of 2.5 grams per liter of retort volume and approximately 10 w/o of the silicon charge were used as the pack materials.

Each pack contained four bend test specimens and three oxidation coupons of V-60Cb-1Ti. The oxidation coupons were about 1 inch square and the bend test specimens were about 3/8 x 1 1/2 inch. All of the material was approximately 22 mils thick before coating.

The relationship between coating thickness, time, and temperature may be seen in Figure 2. Coating growth rate increases with increasing temperature, achieving a thickness of 4.25 mils after siliconizing at 2200°F for 15 hours. The microstructures of some of the coatings may be seen in Figures 3 to 10. All of the specimens examined contained cracks or similar flaws that were normal to the coating-base metal interface. It is of interest to note, however, that only the thickest coatings contained laminar cracks in addition to the normal cracks. The laminar cracking may be seen in Figures 6, 7, and 8. The origin of these cracks is unknown, and they may very likely have been introduced during metallographic preparation. However, their presence indicates a coating weakness which is substantiated in bend test studies that were performed on similar specimens. This work will be described later in this report.

Table I

PACK SILICONIZING RUNS THAT WERE MADE TO DETERMINE THE DEPENDENCE OF TIME AND TEMPERATURE ON COATING RELIABILITY

Run No.	Temperature, °F	Time, hr	Coating Thickness, mils
19	2150	1	1.5
20	2150	15 3/4	4.0
21	2150	3 3/4	2.0
22	2150	7	2.5
23	2200	15 1/4	4.25
25	2200	7 1/2	2.75
26	2000	16	2.25
27	2000	9	~ 2.0 *
28	2000	3 1/2	1.75
29	2200	3 1/2	~ 2.0 *
30	2200	Double-8	4.5
33	2000	Double-8	---

*Interpolated from data in Fig. 2

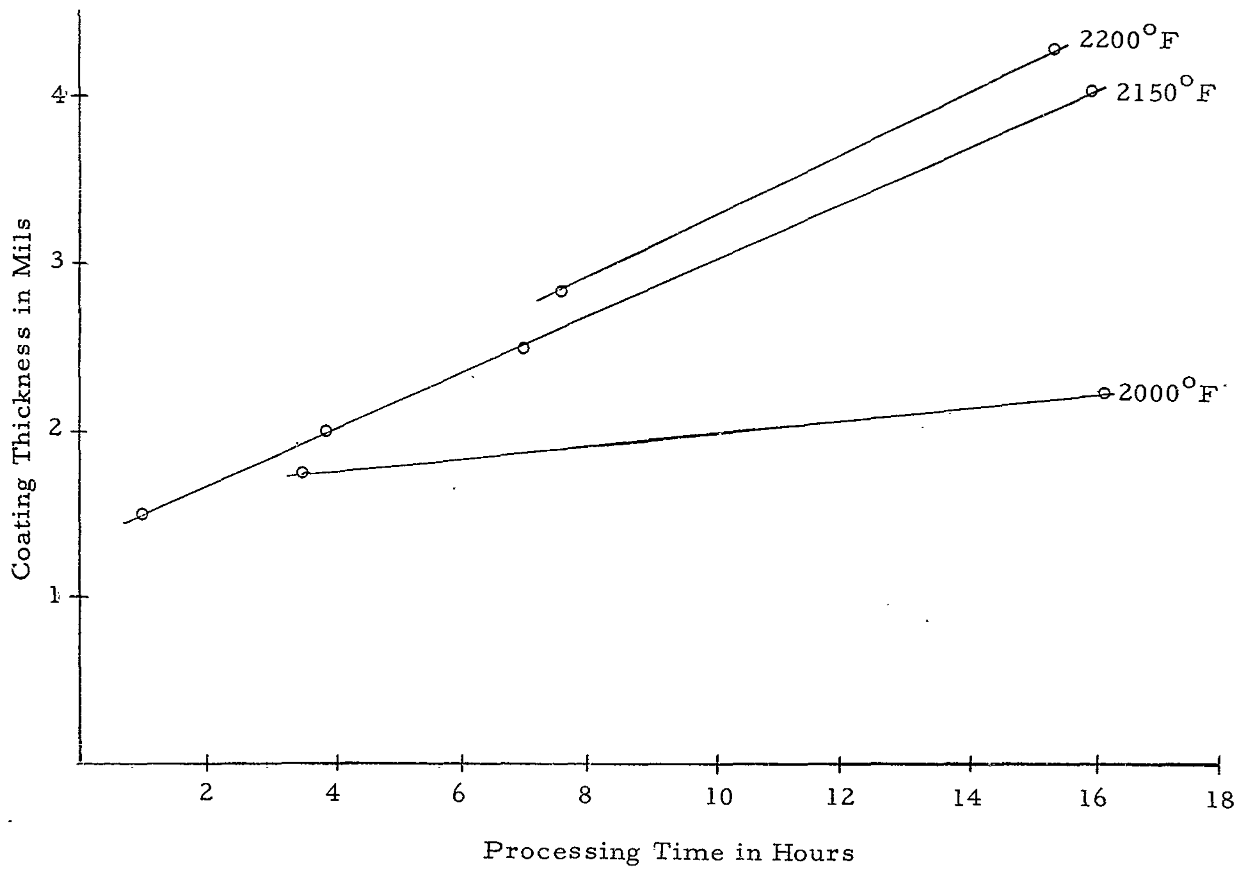


Fig. 2 COATING THICKNESS OF PACK SILICONIZED V-60Cb-1Ti SPECIMENS AS A FUNCTION OF PROCESSING TIME AT 2000°F, 2150°F, AND 2200°F

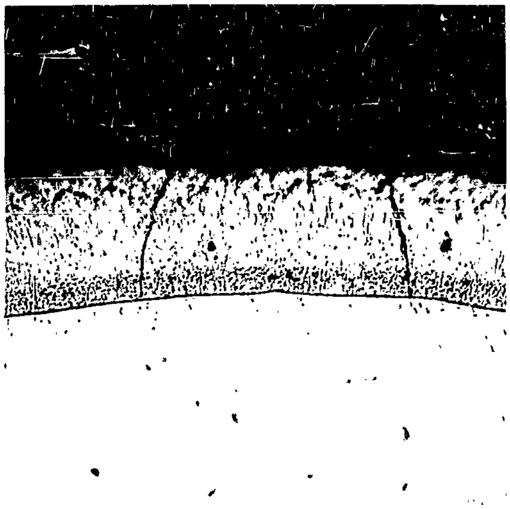


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Fig. 3

V-60Cb-1Ti pack siliconized for 1 hr. at 2150°F. Sample from Run 19.

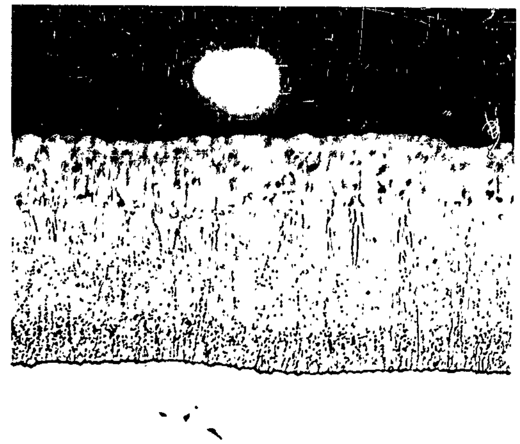

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Fig. 4

V-60Cb-1Ti pack siliconized for 7 hr. at 2150°F. Sample from Run 22.

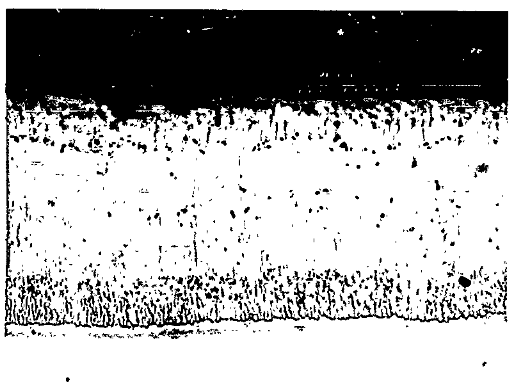

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Fig. 5

V-60Cb-1Ti pack siliconized for 7.5 hr. at 2200°F. Sample from Run 25.

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Fig. 6

V-60Cb-1Ti pack siliconized for 15.25 hr. at 2200°F. Sample from Run 23.

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Fig. 7

V-60Cb-1Ti pack siliconized for 15.75 hr. at 2150°F. Sample from Run 20.

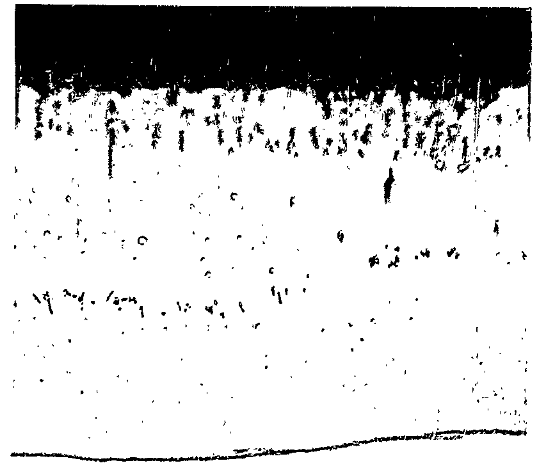

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Fig. 8

V-60Cb-1Ti pack siliconized for two 8-hour cycles at 2200°F. Sample from Run 30.

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Fig. 9

V-60Cb-1Ti pack siliconized for 3.5 hr. at 2000°F. Sample from Run 28.

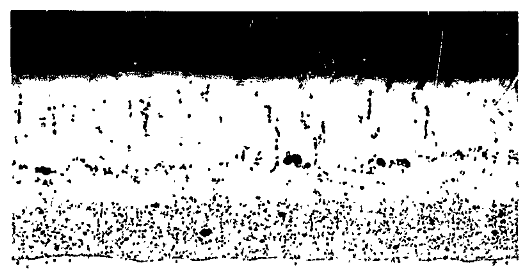

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Fig. 10

V-60Cb-1Ti pack siliconized for 16 hr. at 2000°F. Sample from Run 26.

3. Influence of Coating Thickness on Static Air Oxidation Behavior

The remaining oxidation test coupons were tested in static air at 2200°F. Oxidation data appear in Table II. The specimen having the greatest coating thickness, 4.25 mils, failed after 45 hours when the coating flaked from the base metal during cooling.

The relationship between oxidation life and coating thickness is plotted in Figure 11. Maximum life is observed for coatings having a thickness between 2.25 and 2.75 mils. The very thick coatings apparently fail due to laminar separation during thermal cycling. The mode of failure of the thinner coatings has not been established.

4. Bend Test Studies

The bend test specimens that were prepared in the pack runs previously described were used to determine coating adherence and protective capability after deformation at room temperature.

Coating adherence was determined by subjecting coated bend test specimens to 3-point transverse loading both prior to and after oxidation exposure. Gross delamination and flaking of the outermost coating layers were observed in the coated specimens from Runs 23 and 30 during elastic bending at stresses below the yield point. Failure occurred on the tension side of the specimens. These specimens had the thickest coatings: 4.25 and 4.50 mils, respectively.

Specimens from the other runs were deformed until slight plastic deformation occurred and were then oxidized in static air at 2200°F for times ranging between 10 and 70 hours. Failure, due to oxidation, occurred only in the specimens from Run 20, which had a coating thickness of 4.0 mils. The remaining samples except those from Runs 27 and 28 were brittle after oxidation exposure, indicating coating permeability, apparently as the result of cracks introduced during plastic straining. The specimens from Runs 27 and 28 were capable of sustaining plastic deformation after exposure. These specimens had relatively thin coatings.

Table II

STATIC OXIDATION LIFE OF PACK SILICONIZED

V-60Cb-1Ti at 2200°F

Run	Coating Thickness, mils	Oxidation Life, hr	Type of Oxidation
19	1.5	82.5	general
20	4.0	7	general
20	4.0	43	general
21	2.5	66.5	general
22	2.5	468	edge
23	4.25	45	coating flaked off when cooled for inspection
25	2.75	>502	-
26	2.25	267.5	edge
26	2.25	>307	-
27	2.0	152	general
28	1.75	80	general
29	2.0	>203	-

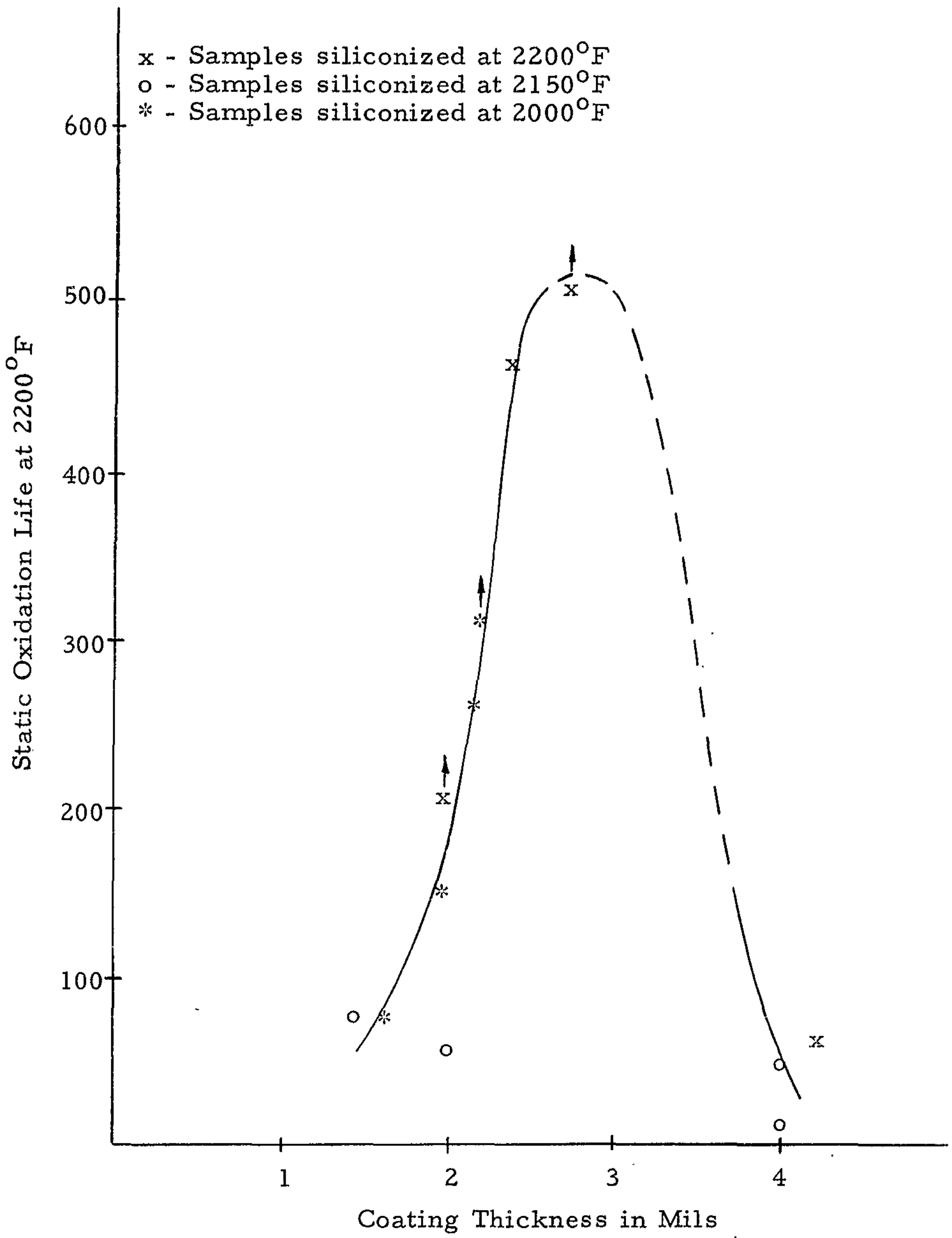

Fig. 11 STATIC OXIDATION LIFE OF PACK SILICONIZED V-60Cb-1Ti TREATED AT 2000°F, 2200°F AS A FUNCTION OF COATING THICKNESS

From these results it appears that the specimens with thicker coatings have a much lower bending stress tolerance. More investigations of this behavior are in progress.

B. Dynamic Atmosphere Oxidation Studies

The oxidation behavior of siliconized vanadium-base alloys is also being examined by direct exposure to an oxygen-hydrogen flame. At present, only preliminary work has been done to establish torch conditions and specimen suitability.

For these tests, one torch was used with has flow rates of 60 cfh oxygen and 120 cfh hydrogen. Specimen temperatures were adjusted by varying the specimen-to-torch distance. Temperatures were monitored continuously with a Thermodot radiation pyrometer using an assumed specimen emittance of 0.8. The emittance value was based on an extrapolation from previous data obtained at lower temperatures. Temperatures were also checked periodically with two disappearing filament optical pyrometers.

The specimens which were used for testing were 1/2 inch wide by 3 inch long portions of 0.050 inch thick sheet. These were self-supported, being rigidly clamped at the bottom The torch was ignited and adjusted before impingement of the flame on the test specimens. Heat-up time was less than 10 seconds, the flame producing a constant temperature zone on the specimens about 3/4 inch long x 1/2 inch wide.

Uncoated control samples were oxidized at 2500° and 2700°F. Failure which resulted in complete "burn through" of the specimens occurred in 13 and 5 seconds, respectively. These times include the heat-up time. Three coated specimens were tested with the following thermal cycles:

Sample 24a	Sample 24b	Sample 31a
35 sec at 2800°F	60 sec at 2800°F	30 min at 2550°F
35 sec at 2800°F	60 sec at 2800°F	30 min at 2500°F
35 sec at 2800°F	180 sec at 2800°F	30 min at 2550°F
45 sec at 2600°F	1 hr at 2700°F	30 min at 2500°F
15 sec at 3200°F	15 sec at 2700°F	

Samples 24a and 24b were used primarily to establish torch distance-temperature relationships. After 4 cycles, the torch-to-specimen distance for sample 24a was decreased to 2 1/2 inches which resulted in melting of the alloy. The temperature at which melting occurred was not recorded but was in excess of 3200°F.

After 4 cycles, including 1 hour at 2700°F, Sample 24b contained fine visible cracks at one edge. During subsequent heating, a small growth was observed in this area and the test was terminated. Both Samples 24a and 24b had a 4.25 mil coating.

Sample 31a developed a small edge defect after four 30 minute cycles at 2500-2550°F. The test was terminated to examine the defected region.

Additional torch tests are currently being made on the 1/2 inch wide coated sheet specimens and similar tests are planned for coated 2 inch square sheet panels.

C. Additional Studies

Other effort on this program is being expended in the following areas:

(a) Coating of tensile test specimens for mechanical property evaluations

(b) Development of a slurry coating process

(c) Coating of vanadium alloy test specimens for other organizations.

Coated tensile test specimens of both the V-60Cb-1Ti and V-20Cb-4Ti-1Zr-0.075C alloys have been prepared. These will be tested in air at temperatures between 1800° and 2200°F to determine tensile and stress-rupture properties.

Some work has been initiated on further study of the Ag-Si slurry coating process reported previously to determine whether such coatings offer a technical advantage over pack siliconized coatings and provide a more versatile coating technique. Preliminary work has attempted to find a suitable binder to replace the flame spray method of applying the coating powders.

Tensile bars of the V-60Cb-1Ti alloy have been pack siliconized for the Boeing Company and a series of oxidation coupons, bend, and tensile test specimens of both the V-60Cb-1Ti and V-20Cb-4Ti-1Zr-0.075C alloys have been coated for NASA, Langley Research Center. This work is being co-ordinated under Contract NOw 62-0101-c, "Pilot Evaluation of Vanadium Alloys". Results of these evaluations of coated specimens will be presented in reports issued under NOw 62-0101-c, and pertinent information will also be discussed in reports under this coatings program.

III. SUMMARY

Pack siliconizing process variables have been studied for silicide base coatings on 0.020 to 0.30 inch thick sheet of the alloy V-60Cb-1Ti. The effect of processing time on coating thickness has been investigated over the temperature range 2000° to 2200°F Coating growth rate increased with time and temperature, attaining a thickness of 4.25 mils after siliconizing at 2200°F for 15 hours. Static air oxidation tests at 2200°F showed that maximum coating life (at least 500 hours) occurs in coatings having a thickness in the range of 2.25 to 2.75 mils. Bend test studies at room temperature showed that the thick coatings (4 to 4.5 mils) failed by gross delamination of the outer layers during elastic bending. Thinner specimens were capable of tolerating plastic deformation of the coated sheet, and subsequent oxidation tests indicated that some of the deformed coatings were protective up to 70 hours.

Dynamic atmosphere testing of 0.050 inch thick coated V-60Cb-1Ti sheet was initiated, using direct exposure to an oxygen-hydrogen flame. These preliminary efforts were devoted to establishing torch conditions, specimen geometry, etc. Other work during this reporting interval included the coating of sheet tensile specimens for elevated-temperature mechanical property evaluations, a study of binders fur use with a sliver-silicon slurry coating process, and the coating of vanadium alloy specimens for evaluations at other organization.

IV. FUTURE WORK

The experimental work described in the preceding sections will be continued. Major efforts will include room-and elevated-temperature mechanical property evaluations of siliconized sheet specimens of V-60Cb-1Ti and a study of oxidation behavior of coated specimens under various conditions of applied stress and atmospheric velocities and pressures. This work will include an evaluation of coating performance at greatly reduced pressures, using a controlled leak rate dynamic system to provide pressures in the range of 0.1 to 100 mm Hg. Short-time tensile and 4 to 8 hour stress-rupture or creep data will be obtained on coated specimens in 2000°F air.

Limited efforts will be devoted to the development of a slurry coating process based on silver-silicon, previously studied under Contract NOw 61-0806-c. These studies will be concerned primarily with the evaluation of binder materials and heat treatments to produce adherent coatings which can be applied by dipping or painting followed by elevated temperature reaction with the vanadium-columbium alloy base. Other work will include the pack siliconizing of test specimens for evaluation under the data-exchange program.

V. LOGBOOKS AND PERSONNEL

Data for this report are recorded in ARF Logbooks C-13280, C-13531, and C-13294.

The following personnel have been the principal contributors to the planning and execution of this work.

F. C Holtz - Project Leader
L. I. Kane - Technical Assistant
J. J. Rausch - Senior Metallurgist
R. C. Vanderjack - Project Technician

Respectfully submitted,

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